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MAGNETIC COLLOID MEDIATED RECOVERY OF CADMIUM IONS FROM AN AQUEOUS SOLUTION USING A FLOW-THROUGH HYBRID FIELD-GRADIENT DEVICE

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ABSTRACT

A flow-through hybrid magnetic field gradient device that uses polymer-coated magnetic particles as a mobile solid affinity phase was developed for the removal of cadmium ions from an aqueous solution. The device consists of a horizontal glass tube rotating about its axis, with repeating hybrid magnetic units positioned along the outside of the tube. Two arrangements for the magnetic units were used. In the first configuration, four hybrid pairs were distributed axially—each pair consisted of an alternating current carrying solenoid, followed by four azimuthally distributed permanent magnets that rotate with the chamber. In the second configuration, all four solenoids were placed first, followed by the four sets of four azimuthally distributed permanent magnets. The polymer-coated (ion exchange resin) magnetic particles and the cadmium-ion containing solution flow concurrently through the tube. The azimuthally flowing alternating current in the solenoid

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introduces axial and radial forces as well as a torque on the magnetic particles, permitting more contact between the particles and the surrounding solution, and allowing cadmium ions to absorb efficiently on to the particle surfaces. The particles are then immobilized on the chamber walls, while the depleted cadmium solution flows through the end of the tube. The 1–10 μm diameter magnetic particles with iron oxide nanoparticles embedded within a quaternary ammonium cellulose matrix (MagaCell-Q, Cortex Biochem, San Leandro, CA) at a starting concentration of 0.5 mg particles/mL were used as the mobile solid support. The feed consisted of a 10.0 mg/L cadmium sulfate solution, at a flow rate of 35 mL/min. The depleted cadmium solution was collected and used as feed for a second stage, where it contacted with a fresh batch of magnetic particles. This was repeated for a third stage. For the first arrangement, the Cd(II) concentration at the exit of the third stage dropped to 0.97 mg/L. The second configuration produced a final Cd(II) concentration of 0.68 mg/L. For the first arrangement, three stage processing resulted in an overall $\sim 90\%$ w/w cadmium removal, and the second configuration removed $\sim 94\%$ of the original cadmium ions. While the specific system studied here consists of removal of cadmium ions from an aqueous solution, the general principle of magnetic colloidal separation using this flow-through device can be exploited for other environmental and biotechnological applications where large volumes of sample must be processed with high capture efficiency.

INTRODUCTION

The US Environmental Protection Agency (EPA) classifies cadmium as one of priority pollutant heavy metals. Sources of cadmium in water supplies include wastes from battery manufacturing, mining, and metal finishing industries, and contaminated groundwater from hazardous waste sites (1). For wastewater with high metal ion concentrations, such as that generated during the manufacture of alkaline storage batteries, precipitation processes are most common and economical for reducing contaminant concentrations (2). In chemical precipitation, hydrogen sulfide is used widely as a reactant (3). However, the addition of hydrogen sulfide has major drawbacks, including the fact that the recovered cadmium sulfide cannot be recycled directly (4).

For removal of metals present in trace concentrations, the most common methods are adsorption on solid supports such as ion exchange resins, metal



oxides, and activated carbon (5). Huang and Ostovic (6) used three brands of commercially available activated carbons as the adsorbent for cadmium ion removal. They have reported a strong increase in the magnitude of cadmium removal as the pH of the system is increased. In addition, they have shown that the increase in carbon to cadmium ratio increases the equilibrium adsorption capacity and initial removal rate (6).

An alternative technique of trace metal ion recovery is the utilization of magnetic particles with a coated absorbent layer such as octyl(pentyl)-*N,N*-diisobutylcarbamoyl-methylphosphine oxide and tributyl phosphate, amines, phosphinic acid, and silicotitanates (7). The advantages of using magnetic particles as the solid phase support include the high surface area available for absorption, avoidance of channeling effects common in packed beds, and the ability to immobilize the target particles at specific locations by application of external magnetic field gradients. Nunez et al. report the successful use of ferromagnetic particles coated with octyl(phenyl)-*N,N*-diisobutylcarbamoylmethyl phosphine oxide diluted by tributyl phosphate for the recovery of americium and plutonium from nuclear waste solutions (8). Similar work has been done on the recovery of cadmium and zinc using extractant-coated (Cyanex-273 and Cyanex-301) magnetic microparticles (9).

The objective of this paper is to demonstrate the feasibility of a newly developed flow-through hybrid magnetic-field-gradient device (10) as an alternative for the removal of low concentration hazardous metal ions from a solution. Potential advantages of this device include the ability to process large volumes of feed solution, efficient mixing of the magnetic particles in the solution, and the ability to use large (micron-sized) magnetic particles which can be mobilized using readily available Al–Ni–Co magnets.

MATERIALS AND METHODS

The separation vehicles are 1–10 μm in diameter (with 75% by number between 2–4 μm) magnetic particles (MagaCell-Q, Cortex Biochem Inc., San Leandro, CA) consisting of iron oxide nanoparticles embedded within a quaternary ammonium cellulose matrix. The polymer serves as an anion exchange resin for the removal of anionic complexes of cadmium with sulfate ions. Cadmium sulfate was obtained as an ACS grade salt from Aldrich Chemical Company (St. Louis, MO). All solutions were prepared using single distilled reverse osmosis water that was passed through a four cartridge Millipore “Milli Q” system (APS Water Services, Inc., Van Nuys, CA) until its resistivity reached 18 $\text{M}\Omega\text{ cm}$. Metal compositions were obtained using flame atomic absorption spectrophotometry (Perkin–Elmer 1100B AAS, Perkin Elmer, Shelton, CT).



Equilibrium Data

Equilibrium data, quantifying the specific adsorption capacity of the magnetic particles were established using a series of batch experiments, and are shown in Fig. 1. Since the device is operated in a concurrent mode, this equilibrium data is useful for calculations of single stage efficiencies. At pH ~ 7 , the maximum adsorption capacity for cadmium ions using MagaCell-Q, is ~ 4.2 mg/100 mg of magnetic particles. The absorption curve fits the classic Langmuir behavior with an equilibrium constant K of 2.98 (mg Cd/100 g solution) $^{-1}$.

Experiment

The experimental device consisting of a 2.0 cm internal diameter, 1 m long, axially-rotating horizontal glass tube, with four axially located repeating hybrid magnetic units, is shown in Fig. 2. In a typical experiment, the chamber is first filled with approximately 375 mL of distilled water. Two hundred milliliters of the cadmium sulfate solution and 200 mL of the magnetic particle-containing solution (particle concentration of 0.5 mg/mL of solution) are driven into the chamber using peristaltic pumps through a rotary coupler. The chamber rotates about a horizontal axis at 25 rpm. An alternating current (12 V, 10 A, 60 Hz) is passed through the solenoids. The supernatant flowing out through the end of the

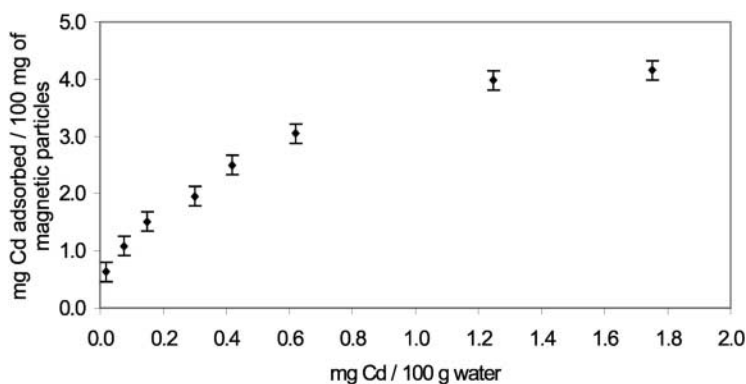


Figure 1. An equilibrium curve constructed for demonstrating Cd(II) adsorption by MagaCell-Q. Magnetic particles are added to a bottle containing cadmium sulfate solution (pH ~ 7.0), and shaken continuously for 5 min. The mixture was then allowed to sit for 1 hr, and samples are withdrawn from the supernatant. Sample concentrations are assayed using an atomic absorption spectrophotometer.



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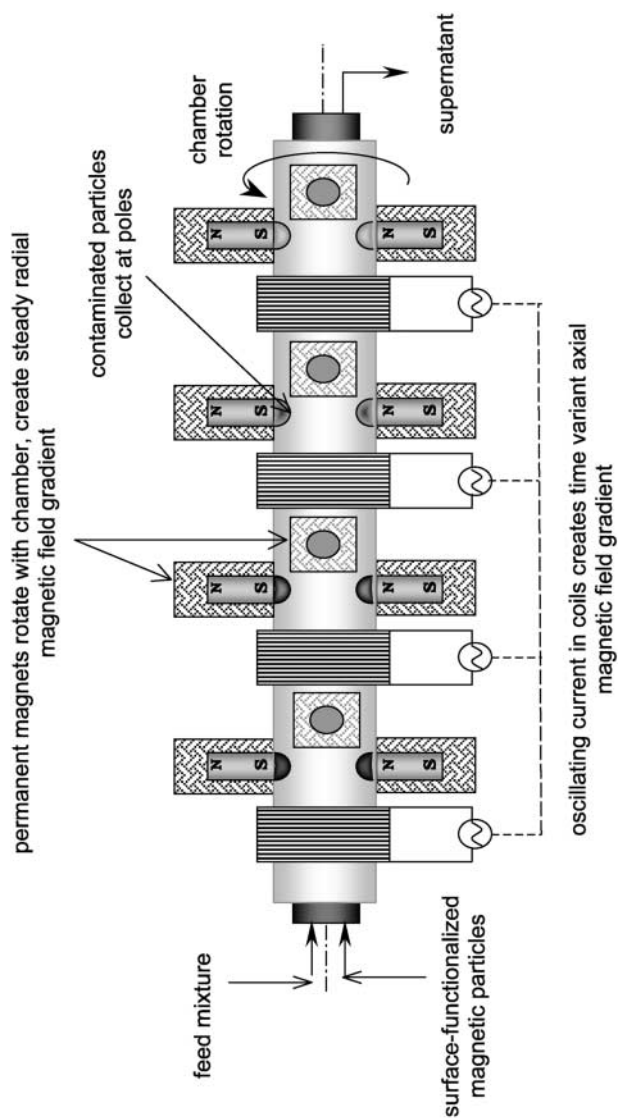


Figure 2. Schematic representation of the 2.0 cm internal diameter 1 m long axially rotating horizontal chamber with four repeating magnetic units (arrangement 1). Each unit consists of a stationary alternating current solenoid surrounding the chamber followed by two pairs of azimuthally distributed permanent magnets that rotate with the chamber.



chamber is collected continuously. When the appropriate liquid volume is collected, the flows of the cadmium sulfate solution and magnetic particle suspensions are interrupted and the total volume of feed that has entered the chamber is recorded. The exit of the chamber is then opened, and 500 mL of distilled water is pumped through, flushing out all of the supernatant. This material is added to the already collected liquid. The concentration of cadmium ions in the supernatant is then measured and multiplied by the total volume to obtain the total mass of cadmium in the supernatant.

The permanent magnets are then removed and distilled water is allowed to flow through the chamber. The cadmium ion containing magnetic particles that had been immobilized at the chamber walls are now resuspended into the chamber and driven out from the other end by the bulk flow. These magnetic particles are designated as the suspension from the pole region. The cadmium ions were stripped from the magnetic particles using a low pH nitric acid solution. However, the highly acidic extract solution proved difficult to analyze using AAS. Thus an indirect, mass balance approach from a knowledge of the feed and supernatant concentrations and volumes are used to calculate the amount of cadmium attached to the magnetic particles in each experiment.

The experiment was operated in a three-stage mode. The feed concentration used for the second stage was determined to be the total amount of cadmium ions in the supernatant collected from stage one, dissolved in the same volume of water as the feed volume for the first stage. A similar procedure was used to generate the feed for the third stage.

EQUIPMENT OPERATING CONDITIONS

Bounds to the flow rates available for successful operation of this equipment are first obtained by calculating the combined influence of the permanent magnet and the flow on the trajectory of a single magnetic particle. As a first approximation for the successful immobilization of a magnetic particle, the time taken by the particle to travel radially over one tube radius must be less than the time taken to travel axially in the chamber over a distance corresponding to the diameter of the permanent magnet (Fig. 3). The trajectory of a single magnetic particle under the influence of a magnetic field gradient, gravity, and viscous drag is governed by:

$$m_p \frac{dV_p}{dt} = F_G + F_D + F_M \quad (1)$$

where m_p is the mass of the particle, V_p is the particle velocity, F_G is the force on the particle due to gravity, F_D is the drag force, and F_M is the magnetic force on the particle. Since slow chamber rotation emulates a low gravity environment, the



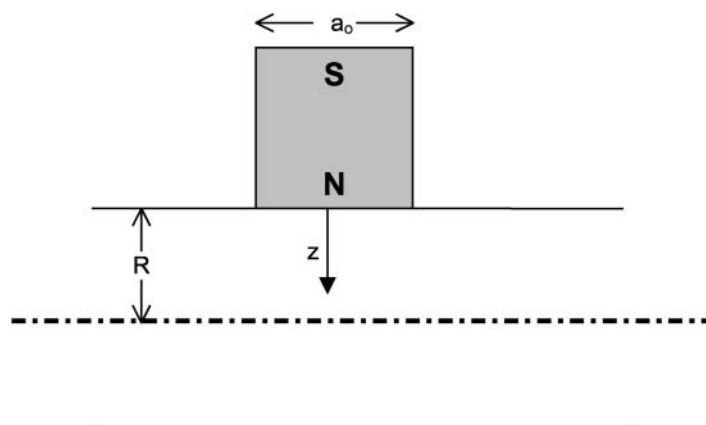


Figure 3. Illustration of the calculation used to determine the maximum flow rates available without losing magnetic particles to the flow. The time required for a particle to move radially over a length equal to one tube radius should be less than that required to move axially over a distance corresponding to the permanent magnet diameter.

first term F_G is ignored. For a spherical particle of diameter D_p , these forces can be expressed as:

$$F_D = 3\pi\mu D_p(V_p - V_L) \quad (2)$$

$$F_M = \frac{\pi D_p^3}{6} M_s \cdot \nabla B \quad (3)$$

Here V_L is liquid velocity, μ the liquid viscosity, M_s the particle saturation magnetization, and B the applied magnetic field. Since the liquid has no radial velocity, the flow rate does not affect the radial motion of the magnetic particles towards the tube wall. For determining axial motion of the particles, the particle axial velocity is taken to be the same as the maximum liquid velocity, calculated using laminar, unidirectional flow in a cylindrical tube.

The component of the applied magnetic field, B , along the axis of the permanent magnets is

$$B(z) = B_s \left[1 - \frac{z}{\sqrt{(a_0^2 + z^2)}} \right] \quad (4)$$

where B_s is the magnetic field at the surface of the magnet, z is the distance away from the surface of the permanent magnet, and a_0 is the radius of the permanent magnet.



The saturation magnetization of the magnetic material within the particles is 10 emu/g. The magnetic particles have an overall density of $\sim 1.9 \text{ g/cm}^3$, and have 12% w/w iron oxide loading. The radius of the permanent magnet is 1.2 cm, and the measured field, B_s , at its surface is 1.5 kG. Using these parameters, the radial position of 1.0, 5.0, and 10.0 μm spherical particles as a function of time, released at the chamber axis at $t = 0$ are shown in Fig. 4. The magnetization of particles varies as the cube of its diameter, while the drag force is proportional to the diameter. Thus, the larger particles arrive at the chamber walls in shorter time than the smaller ones. Using these results, the upper bound on the volumetric flow rate available for each size particle is shown in Fig. 5. For example, the maximum flow rates that can be used for 1.0, 5, and 10.0 μm diameter particles are 21.7, 126.7, and 506.8 mL/min respectively. Note that if any particles are not captured by the first set of permanent magnets, they are acted upon by the remaining three. In our experiments we have restricted the volumetric flow rate to 35 mL/min, and see a negligible concentration of magnetic particles in the supernatant.

In order to determine the breakthrough characteristics of the device, in the experiments, the chamber is filled first with distilled water. A 5.0 mg/L cadmium sulfate solution is then introduced as the feed. The solution flowing out of the end of the chamber is collected in 20 mL aliquots. Each sample was then analyzed

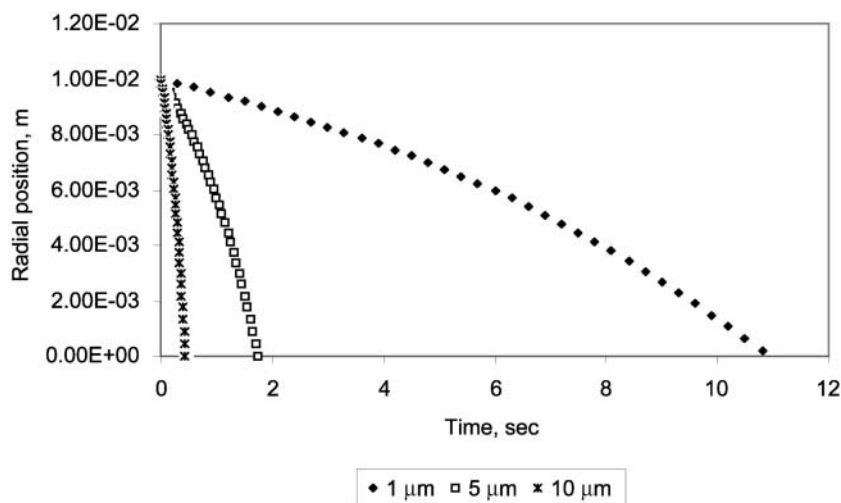


Figure 4. The position of a single particle, launched at zero velocity at the intersection of the tube and permanent magnet axes. The larger particles arriving at the tube wall are faster than the smaller particles. Since there is only axial velocity in the tube (along with an azimuthal velocity corresponding to rigid body rotation), the particle trajectory in the radial direction is unaffected by the flow.



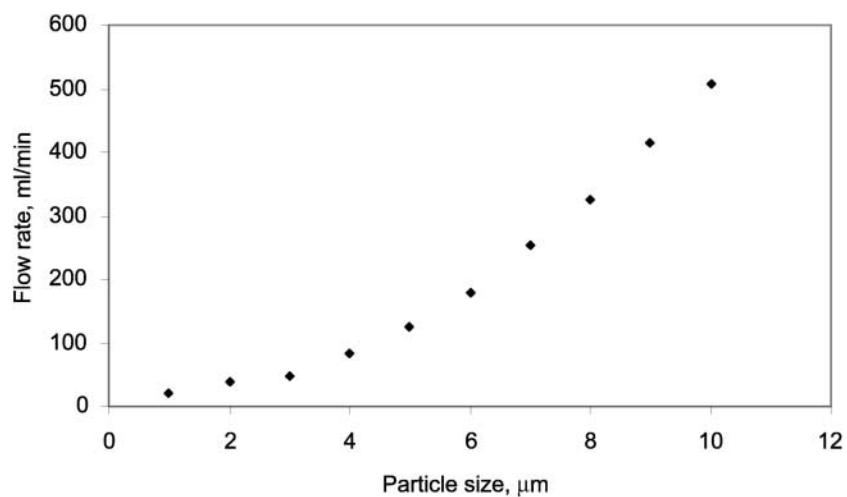


Figure 5. The maximum flow rate that can be sustained in the apparatus without loss of particles. The operating regime consists of the region below the plot.

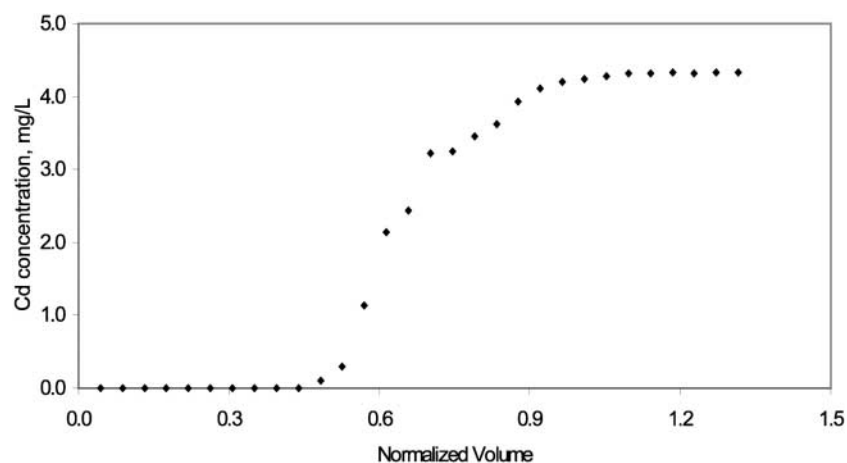


Figure 6. Cadmium ion concentration vs. normalized volume (volume of liquid flowing in supernatant/tube volume) for solution flowing out of the end of the chamber. Breakthrough occurs at 0.7 chamber volumes. The device is operated at steady state conditions in these experiments.



and results are displayed in Fig. 6. This figure clearly demonstrates that once $\sim 70\%$ of the chamber volume of solution is withdrawn, breakthrough is achieved and the device subsequently operates at steady state conditions. In our experiments, we used a total feed volume that is 1.5 times the chamber volume, ensuring that the supernatant is a “steady-state” sample. In addition, a buffer volume corresponding to the chamber volume was used to drive out all the remaining cadmium ions after cessation of feed flow.

Two arrangements of the device were used in the experiment to determine cadmium ion recovery using magnetic particles. In the first configuration shown in Fig. 2, each magnetic unit consists of the solenoid and four azimuthally distributed permanent magnets. The first unit was located 40.0 cm downstream from the entrance of the chamber followed by three others positioned axially downstream, each separated by 4 cm. The chamber rotation simulates a low gravity environment, severely attenuating any sedimentation of non-neutrally buoyant magnetic particles, thus promoting good particle–target contact throughout the chamber volume. The oscillating magnetic field gradient produced by the solenoid introduces translational and rotary particle oscillations, enhancing mixing. The permanent magnets immobilize the contaminated particles on the chamber walls. The reported cadmium ion concentrations represent an average of five experiments, with three samples withdrawn from each experiment for analysis.

RESULTS AND DISCUSSION

All experiments were conducted at an overall (feed + magnetic particle suspension) flow rate of 35 mL/min. The feed to the first stage consists of 10.0 mg/L cadmium sulfate solution. For the first arrangement of magnets and solenoids, the results are displayed in Fig. 7. Thirty eight percent of the cadmium in the solution, which is removed after one stage reduces the concentration to 6.2 mg/L. The stage efficiency, measured as the percent approach to equilibrium along the operating line, is 51%. A further 45% is recovered in stage 2, lowering the concentration of cadmium ions in the supernatant to 3.4 mg/L. The stage efficiency is 53%. At the exit from the third stage, the cadmium ion concentration is reduced to 0.97 mg/L, and the stage efficiency is 67%. The slope of the operating lines for each stage was determined by calculating the ratio of solution feed flow rate to particle flow rate. The calculated slope is 2.0, and is in good agreement with the experimentally obtained slopes of the operating lines for each of the three stages of 1.94, 1.91, and 1.89 respectively.

For the second arrangement, shown in Fig. 8, the four alternating current solenoids are placed in sequence, followed by the azimuthally distributed permanent magnets that rotate with the chamber. This type of arrangement allows



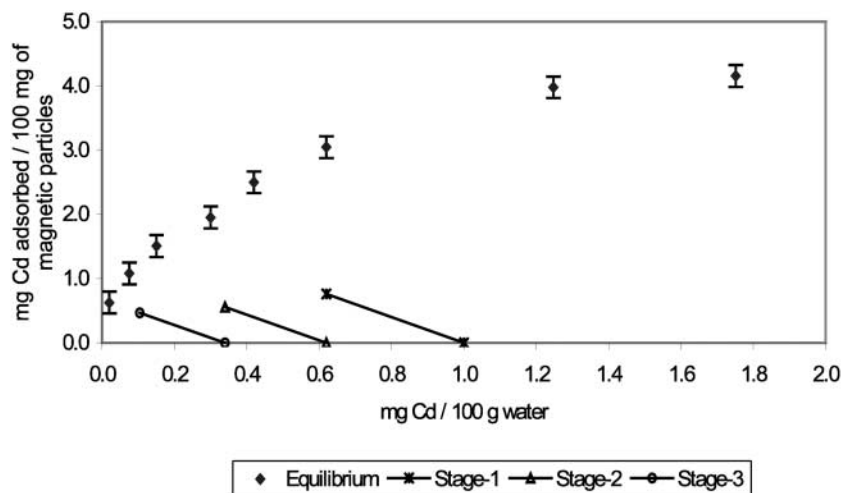


Figure 7. Cadmium ions adsorbed by magnetic particles vs. feed cadmium concentration for the arrangement where each solenoid is paired with permanent magnets. In first stage, 10.0 mg/L cadmium sulfate solution and magnetic particles at 0.5 mg/L concentration were processed and the supernatant collected. For the second and third stages, the supernatant was fresh, magnetic particles were added to each of the feeds.

the particles to stay in intimate contact with the solution for longer times than the previous configuration before being immobilized on the chamber wall. The separation results are displayed in Fig. 9. The supernatant from the first stage contained 5.6 mg/L cadmium ions indicating a 43% recovery. The stage efficiency is 54%. By the second stage an additional 49% of the cadmium ions are recovered, bringing the supernatant concentration to 2.8 mg/L. The stage efficiency is 58%. The cadmium ion concentration at the end of the third stage is 0.68 mg/L. The stage efficiency is 65%. The experimentally measured slopes of the operating lines for the three stages are 1.89, 2.20, and 2.16 respectively, and closely correspond to the calculated value of 2.0.

The stage efficiencies reported here range from 50–60%. Since this is a concurrent operation, increase of the stage efficiency would require longer separation chambers, and would add significantly to the cost of the device. A multistage stage operation is therefore the easiest way to overcome this limitation, and is the route demonstrated here.

The use of ion exchange resin-coated magnetic particles as the separation vehicles offer some advantages over the traditional packed bed scheme. Since all of the resin is on the outside of small diameter particles, they get exposed to the solution, thus overcoming the need for transport of metal ions to the internal pore



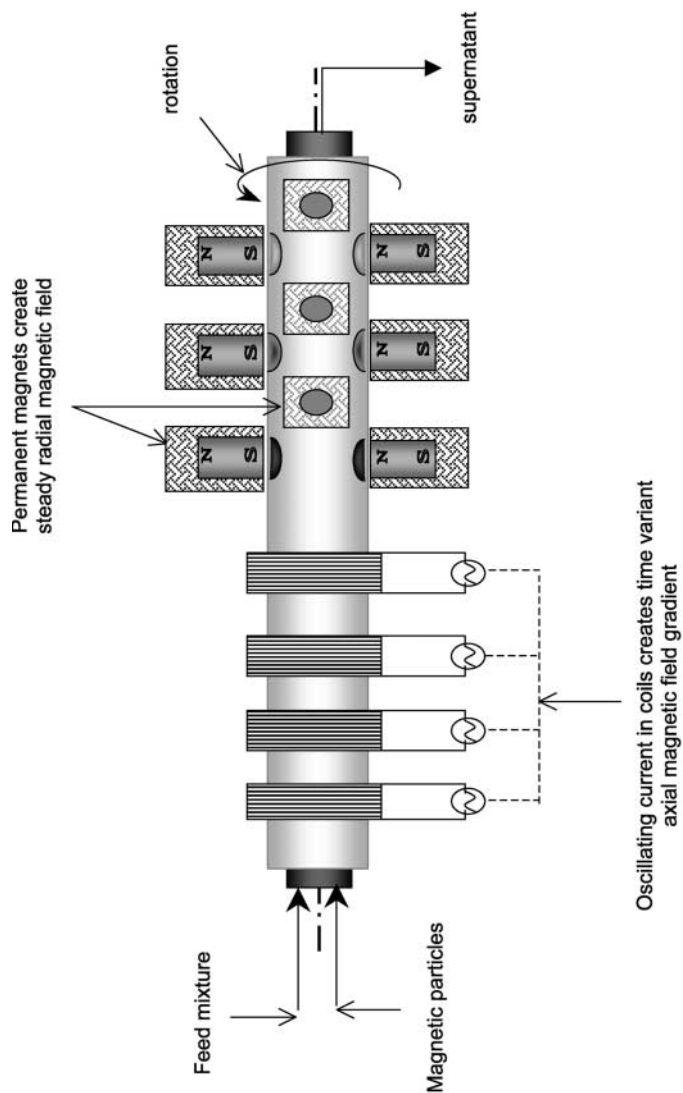


Figure 8. Schematic representation of the 2.0 cm internal diameter 1 m long axially rotating horizontal chamber with four stationary alternating current solenoids surrounding the chamber followed by azimuthally distributed permanent magnets that rotate with the chamber (arrangement 2).

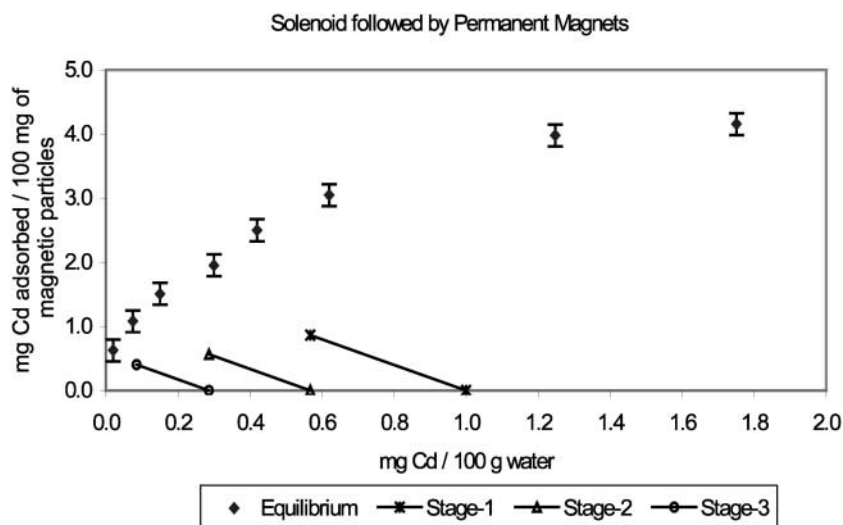


Figure 9. Cadmium ions adsorbed by magnetic particles vs. feed cadmium sulfate concentration for arrangement 2. The final concentration at the end of three stages is lower than that available in the first arrangement, indicating that the enhanced contact time prior to the capture of magnetic particles has an impact on the separation.

surfaces. This feature is important for both the absorption, as well as desorption/resin regeneration cycles. In addition, the concurrent flow of the magnetic particles and the cadmium sulfate solution means that pressure drop required to mobilize the solution through the apparatus are much less than those required for packed beds for the same volumetric flow rate.

While the flow rates available in this equipment appear to be limiting at first, several of these devices can be run in parallel to overcome this. In addition, larger magnetic particles can be used (chamber rotation eliminates gravitational settling; the only disadvantage of using larger particles is the lower specific surface area for ion absorption), allowing for a greater flow rate in any single device. A device similar to this one, where the feed solution consists of a resin-regenerating salt can be used to recover the metal ions and recycle the resin. Thus, this equipment may be suitable for operations in the electroplating industry, where precious metals need to be recovered from plating solutions where the ion concentrations are low.

The EPA regulatory requirement is a concentration below 1 mg/L for safe discharge of cadmium into a publicly owned treatment system (11). Starting with a concentration of 10 ppm, three stages are required in this device to drop the concentration to the EPA acceptable level. This device thus represents a viable



technique for metal ion removal. Indeed, depending upon the nature of the polymer coated on the surface of the magnetic particles, a wide range of contaminants can be removed, and if necessary, recovered while regenerating the resin.

CONCLUSIONS

The newly developed flow-through hybrid magnetic field gradient device has been used for cadmium ion recovery from a cadmium sulfate solution using magnetic-ion-exchange-resin particles as a mobile support. Two arrangements of the device were employed. In the first, four pairs of alternating current carrying solenoids and azimuthally distributed permanent magnets were arranged axially along the tube. In the second, all four of the solenoids were mounted first, followed by the permanent magnets. For the first arrangement, a three-stage operation allowed the cadmium ion concentration to be reduced from 10.0 to 0.97 mg/L. For the second arrangement, a three-stage operation reduced a 10.0 mg/L feed concentration of cadmium to 0.68 mg/L. This represents an over all cadmium ion removal of ~90 and 94% respectively. The device is therefore well suited for the removal of toxic metal ions from a solution.

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